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HIGHLY CONCENTRATED EMULSIONS OF THE OIL-WATER TYPE

VII. STABILITY OF EMULSIONS PROTECTED BY SODIUM OLEATE

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(Tables and figures referred to in the text are appended. Numerals in parentheses refer to the bibliography.)

In the present article we shall follow the previously developed plan (1). Each of the principal processes of aging emulsions -- coalescence and separation -- will be studied separately, so that their relationship may finally be established. In this it is natural to expect that a number of fundamental rules established by us for gelatin emulsions (1, 2, 3) will also apply to oleate emulsions. However, in view of the essential differences in structure and properties between the two emulsifiers, we must expect essential differences in their behavior as well.

Coalescence

As was pointed out in the foregoing article (4), protective films of oleate vary according to the conditions of synthesis. First of all, condensed and then expanded single layers are formed, while polymolecular films form only when special conditions are maintained.

- 1 -

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When we consider emulsions with monomolecular protective layers, it must be expected that coalescence in systems with expanded and condensed single layers will fail to proceed uniformly. Since in the first case the protective films are unsaturated as far as adsorption is concerned, according to our ideas of the coalescence mechanism a limited coalescence will take place. This process is characterized by the fact that its rate in time drops almost to zero, while the dispersed oil is practically unseparated in the form of a macro-layer. The changes taking place in the system during its aging amount to the well-known diminishing of the degree of dispersion and increase of the density of the cover by the emulsifier at the interface between phases.

For a similar limited coalescence we introduced (1) an equation connecting the mean diameter of the particles \bar{d}_m with the time of aging of system τ :

$$\bar{d} - \bar{d}_0 = (\bar{d}_m - \bar{d}_0) \frac{\alpha' \tau}{1 + \alpha' \tau}$$

where \bar{d} is the mean diameter of the drops of oil at time τ , \bar{d}_0 is the initial mean diameter ($\tau = 0$); \bar{d}_m is the mean diameter with $\tau \rightarrow \infty$; α' is a constant.

Actually, Harkins and Beeman (5) and subsequently Fisher and Harkins (6) established rules for an emulsion of benzene in water with expanded oleate films ($\sim 46 \text{ \AA}^2$) in the process just described. This is illustrated by the curves in Figure 1, constructed according to the data of Fisher and Harkins. The dotted curve $O(\tau)$ represents the change in time of the area occupied by a soap molecule in an adsorption layer. The curve $\bar{d}(\tau)$ represents the change of the mean diameter \bar{d} of oil drops also as a function of the time of aging of the systems. It is evident from the drawing that as the drops increase in size the density of the soap films also increases to the point of a state of condensation (19.5 \AA^2 for 10 days of aging).

We used these data for checking equation (1) graphically. In the system the equation $\frac{\bar{d} - \bar{d}_0}{\bar{d}_m - \bar{d}_0}(\tau)$ is a straight line and, as can be seen from Figure 1, the experimental points provide the required relationship. We note in this connection that for d_0 in the calculations we took the value d throughout 16 hours of aging, since the magnitudes for the earlier period (up to 16 hours) vary irregularly. Consequently, the time reading is also taken from 16 hours.

Thus, our equation for the coalescence energy is justified for emulsions protected by expanded films of oleate just as for gelatin systems. We may conclude from this that the coalescence mechanism fundamentally does not depend upon the nature of the emulsifier, provided certain general conditions are observed (adsorption saturation of the layer).

Hitherto we have been considering a spontaneous process. However, we may come to the same ultimate result in the change of adsorption layers by another method, that is, by subjecting a freshly prepared emulsion to a weak random mixture by hand (shaking in a container). In this case the emulsion begins to break down rapidly at first, and then slowly and after about half an hour the oil phase is distinguished by sparse drops. The remainder comprises a much more stable system. If we make an analysis of the initial system and the remainder after mixing, we can readily discover the essential nature of the process.

We conducted such an investigation for three emulsions containing benzene, oleate, and water, prepared in the usual way (4). The dispersion analysis and determination of soap adsorption was done by the methods described in the preceding article. The results are collected in Table 1.

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As can be seen from the table, the necessary process is accompanied by a considerable compression of the interface, chiefly due to a partial breakdown of the emulsion, while the protective oleate films are contracted almost to the point of condensation.

Passing now to a system with condensed protective oil films, it is possible to expect that their aging will take place in a quite different form.

Similarly, it appears that the dispersion of a similar type of emulsion remains constant for a long time, according to Table 2, where the collected results of dispersion analysis of three emulsions, contained in test tubes with ground-glass stoppers (33 mm in diameter) for about a month at 14-15 degrees, are given.

Harkins (5) also notes the great stability of such systems. For one of the samples he could discover no appreciable variation in the degree of dispersion after keeping it a year.

However, it is not yet possible to draw conclusions from these observations as to the absence of any coalescence. The latter, as we shall see below, takes place in a very peculiar fashion.

Our emulsions, when stored, display a marked tendency to give off free benzene, which, it stands to reason, is inconceivable without mutual fusion of the separate drops. This breakdown of the emulsions is illustrated by the curves in Figures 2 and 3, where the quantity of released benzene is given on the ordinates in percentages of its initial content in the system, and the aging time in hours is given on the abscissae. The curves in Figure 2 refer to the series of saturated emulsions with diverse concentrations and condensed protective oleate films (see also (4)). The emulsions were kept in test tubes with ground-glass stoppers (diameter, 33 mm), while from time to time the liberated benzene was measured with a measuring cylinder after the emulsion had been thoroughly mixed with a glass rod. The curves in Figure 3 were obtained by the same method for a 12:1 emulsion, uniform amounts of which were kept in cylindrical containers with diameters ranging from 46 to 106 mm. During the escape of evaporating benzene the emulsion containers were placed in a drier over a layer of free benzene.

As can be seen directly from the figures, all emulsions, and especially diluted ones, had broken down considerably. Another fact is very noticeable, that is, the relation of the breakdown rate and the form of the breakdown curves to the diameter of the container.

The breakdown rate generally increases with the circumference of the container in which the emulsion is stored (Figure 3). On the other hand, the breakdown curves in small containers have an S-form, that is, the process here has a somewhat autocatalytic nature. The curves for the large containers are concave in relation to the abscissae during the entire time of study of the aging process.

As it appears to us, these last facts throw light on the breakdown mechanism of emulsions with condensed soap films. Since the rate of breakdown depends upon the size of the emulsion column [in the container], we may assume with great probability that the drops of the oil phase on the surface layer of the emulsion in contact with the external non-polarized medium (originally air, and then the monolayer of benzene) are under much less favorable conditions than those within the contents. Evidently the asymmetry of the conditions of existence of an oil

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drop near the free surface of the system is noticeably distorted, as a result of which the drops here fuse with each other incomparably faster, as well as with the macrolayer of benzene.

This peculiar process is called surface coalescence as contrasted with volume coalescence, the probability of which does not depend upon the spatial position of the dispersed drops.

From this point of view it is also not difficult to explain the autocatalytic nature of certain curves (small containers). Similarly, surface coalescence may take place not only on the edge of the free surface of a system, but also within its volume at the edge of coarse macro-occlusions of air and benzene. The latter are, so to speak, the centers of coalescence, and as they gradually increase in size they in their turn speed up the process. If the free surface of the emulsion is small (small container), the role of similar macro-occlusions becomes relatively greater, as a result of which the breakdown curves acquire an autocatalytic character.

Thus, when protected by condensed soap films, coalescence proceeds very rapidly, especially in the layers near the edge of the emulsion.

Let us note in conclusion that systems with polymolecular protective films have not been investigated intensively by us. It is nevertheless to be noted that they have a still greater stability.

Separation and Expansion

Separation of emulsions and expansion of the films are processes of greatest importance to the theory of the stability of highly concentrated emulsions. With the help of the studies of these processes in previous articles (2, 3) we were able to explain in detail the active function of water when protected by a colloid emulsifier (gelatin). We shall now carry out an investigation along the same plan in the case of emulsions protected by sodium oleate.

We are interested in two basic problems: (1) reversibility of the water bond and the critical state of emulsions, and (2) the nature of the water bond in oleate emulsions.

Preliminary experiments on separation of highly concentrated oleate emulsions in centrifugal fields have already shown that the films formed, as in the case of gelatin systems, are capable of expansion. In other words, here the precipitation of water is also at least a partially reversible process.

We made a detailed study of the kinetics of separation in centrifugal fields of different strength (from 1,000 to 4,000 rpm) and the kinetics of expansion of films for a series of saturated emulsions with condensed protective oleate films (emulsion ratios of 5:1, 16:1, 12:1, 18:1, and 32:1). The syntheses and the detailed characteristics of these systems, as well as the method of measuring the kinetics of separation and expansion, were set forth in previous reports (4, 2).

The results of certain experiments are represented graphically in Figure 4 (on the right is the separation, on the left, the expansion); time T serves as the abscissa, and along the ordinate is given the relative water content in films $\frac{V_0 - V}{V_0}$ in percent.

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The data on the kinetics of separation were used to check the theory of the process we proposed. Since the kinetics of separation are not properly of direct interest for the solution of the problems posed above, we shall limit ourselves entirely to the observation that this check showed a satisfactory correspondence between the theoretical and experimental results.

It is now important for us to determine the minimum water remainder in emulsions in their separation in centrifugal fields without breakdown, and the maximum water content in the films after their saturation expansion. Evidently, the reversible measurements of our systems will also lie within these limits.

It is not difficult to determine the first quantity from the separation curves by extrapolation to $\tau \rightarrow \infty$. This quantity, of course, will diminish with the increase in strength of the centrifugal field. The experiments, however, show that even with relatively little strength (1,000-2,000 rpm) an almost saturated separation is reached.

The second of the indicated quantities (maximum remainder) can be found from the curves for the expansion of the films. However, this method can give a correct result only if there is still a certain remainder of free water after the expansion of the films. And meanwhile the films obtained from highly concentrated systems (18:1 and 32:1), counterabsorb all the separated water. One can suppose, that here the original water content is below the equivalent remainder, and therefore such emulsions should expand in water even without preliminary separation.

Actually, we also observed this for 18:1 and 32:1 emulsions. In this case we completed the following experiment for determining the equivalent expansion.

A test tube was approximately half filled with emulsion. The surface of the emulsion was carefully leveled and the grease was removed from the walls of the test tube. Then the test tube was immersed upside down in water within a larger test tube with a ground-glass stopper. After a quick double turnover of the whole system, the small test tube was filled with water up to the level of the emulsion. At the same time the position of the level was noted in a cathetometer and after 30 to 40 hours of expansion, sufficient to establish an equilibrium, the downward displacement of this limit was measured. Knowing the original concentration of the emulsion, its initial volume in the test tube, and the increase in volume upon expanding, it is not difficult to determine the equivalent content of the bonded water. A diagram of this experiment is given in Figure 5.

In this experiment two methods were used, the method of extrapolation according to the curves of the expansion of the films (emulsions with ratios of 5:1, 16:1, and 12:1), and the capacity just described of the original emulsions to expand, and we were able to establish for a number of systems the maximum amount of bonded water.

Finally, knowing the equivalent values of the water remainder in expansion or separation and the total interface between phases (according to the dispersion analysis data (4)), it is not difficult to compute the thickness δ of the aqueous layers according to the formula

$$\delta = \frac{\Delta V}{S}$$

where ΔV is the equivalent water remainder in the system, and S is the surface of the interface.

- 5 -

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These final results of our experiments are assembled in Table 3.

Thus, Table 3 indicates the area of reversible change of the intermediate water layers in our emulsions and, as can be seen from the table, in the first approximation the limits of this area are not related to the concentration of the systems.

We have already encountered a similar fact in studying the intermediate layers of the continuous phase in gelatin emulsions with the sole difference that there the equivalent films in expanding rose only after their known nonreversible condensation as a result of the single centrifugal quality of the system (2).

However, the essential difference between emulsions protected by gelatin and soap was another matter, in that it lay in the relative position of the critical state. We recall that the latter is reached with such a thickness of the layers of the continuous phase, when with the external mechanical effect (mixing) the emulsion begins to break down with the liberation of dispersed oil in the form of a macro-layer. In the case of emulsions protected by gelatin the critical thickness of the films lies between the upper and lower equivalent values of δ , that is, it falls in the area of the reversible change of films (3). In the case of emulsions with condensed protective oleate films the extraction of water to the point of the minimum remainder does not bring about breakdown of the systems. Evidently the critical thickness of the layers here coincides with or is even somewhat less than the minimum equivalent value of δ . This is also supported by the results of previous work. As was found there, the average thickness of the water layers in which a synthesis was still possible of saturated emulsions with condensed oleate layers is equal to about 900 Å, which corresponds well with the average magnitude of δ_{MIN} in table 3 (1037 Å).

However, if in our systems the critical limit cannot be transcended by the mechanical method (centrifugal effect) another method can be used, namely, that of freezing the water. For this purpose we subjected two systems with ratios of 12:1 and 32:1 to cooling at a constant temperature below zero. A known volume of each of the emulsions was continually stirred with a thermometer, permitting measurements of the temperature of the systems from time to time, while the volume of released benzene was determined by pouring it off in a measuring cylinder.

The results of the experiments are shown in Figure 6 in the form of two pairs of curves: one pair corresponds to the temperature change of the system with time t (T) and the other pair represents the amount of separated benzene in percentages as functions of time, O/O (T).

In considering the curves for t (T) their peculiar form immediately strikes the eye. After following a smooth path in the process of cooling the emulsion from room temperature, the curve takes a sharp jump, the cause of which can be interpreted as the beginning of the supercooling of the system. Then there is a horizontal section of constant temperature, the end of which coincides with complete freezing of the water. On the other hand, from a comparison of both pairs of curves t (T) and O/O (T) it appears that the progressive breakdown of emulsions only starts from the moment of freezing of the known part of the water, and the smaller that part is, the more concentrated the original emulsion is. For a 32:1 emulsion, in which the water films are thinnest (~ 900 Å) the beginning of breakdown practically coincides with the temperature jump in curve t (T), that is, with the beginning of the freezing of the water phase.

- 6 -

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Thus, by quite different means we arrived at the previous value for the critical thickness of the water layer, and consequently for emulsions with condensed soap films it actually coincides with the minimum equivalent thickness indicated by the lower line in Table 3.

It is interesting to note that if the freezing point of the continuous phase of the emulsion is lowered, the temperature limit of the breakdown of the system is changed accordingly. We diluted a 48:1 emulsion (97, 96% by volume) with glycerin three times the volume of the water phase. The glycerin was added in small portions with brisk mixing by an electric mixer, and finally we obtained a very stable and transparent 12:1 emulsion and a continuous phase composition of 25 percent by volume of water and 75 percent by volume of glycerin. For this emulsion we plotted the cooling curve $t(T)$ in Figure 7, along with the corresponding curve for the 12:1 emulsion without glycerin (dilution of a 48:1 system by water).

As is evident from Figure 7, the curve $t(T)$ for an emulsion with glycerin is a steadily falling branch showing cooling to thermostatic temperature. It is evident that the horizontal plateau of curve $t(T)$ and consequently the area of breakdown can be reached only upon much deeper cooling.

Consequently, by adding glycerin to prepared, highly concentrated emulsions protected by soap, nonfreezing systems can be successfully prepared. Ethyl alcohol, acetone, ethylene glycol, and thiodiglycol are not suited to this purpose, nor are salt solutions. All of them, in the absence of glycerin, immediately break down the emulsion.

We still have to determine the nature of the forces which sustain a considerable part of the water phase in a combined state in emulsions with condensed protective oleate films. As was noted before (4), on the border of the section of oil drops an electrical double layer must develop due to the partial dissociation of ion-generating groups of soap molecules, forming a protective film. It is not difficult to demonstrate this by electrophoresis made on a concentrated emulsion.

We carried out an experiment for this purpose, illustrated in Figure 8 (platinum electrodes). If the negative pole in this system is located on the bottom, and the circuit is closed, the water forms the border of the emulsion section; this level, recorded by a cathetometer, remains steady. If the poles are interchanged, the border very quickly drops toward the lower positive electrode -- the emulsion absorbs the water, so to speak. In the subsequent switching of poles the emulsion column is contracted to its former level, but the process is much slower.

Measurements of V , made for an 18:1 emulsion with condensed oleate films with motion of the border of the section below, gave a value of $5.85 \cdot 10^{-4}$ cm/sec for the electrophoretic mobility of the oil drops. This figure shows a good correspondence with the data of other authors (7, 8, 9).

Thus, in discussing the behavior of our systems we must consider the interaction of electrical double layers. From this point of view it is not difficult to understand that spontaneous separation of our emulsions can take place only when diffused ionic atmospheres of the adjacent drops do not yet intersect. In the opposite case, electrostatic repulsion forces arise between the drops of oil which prevents their joining and separating from the water. Separation again becomes possible only under the influence of the external forces of a centrifugal field.

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However, for every force applied externally a condition of equilibrium will ultimately be reached which corresponds to the undistorted state of the diffused atmospheres. This is also the cause of expansion of emulsions protected by soap.

It can be concluded from the above that the maximum equilibrium (expansion) and the minimum equilibrium (separation in centrifugal fields) thicknesses of the water layers corresponds to the undistorted and fully distorted condition of the ion atmospheres of the electrical double layers in an emulsion.

The conclusion that salt solutions prevent emulsions from expanding follows, subject to experimental verification.

Thus, in contrast with gelatin emulsions, where the known part of the water is combined by the osmotic expansion forces of the gelatinous protective layers, in the case of oleate emulsions the water remains combined in the system because of the electrostatic repulsion forces which prevent the oil drops from joining. This is what prevents decrease of the thickness of the water phase layers to the critical amount, and this in turn keeps the system from very swift coalescence and from breakdown.

Thus, on the basis of our investigations we can make a well-supported assertion that the main cause of the stability of highly concentrated emulsions is the active forceful interaction between protective films of oil drops. The nature of this interaction (osmotic or electrostatic) depends upon the nature of the emulsifier, and consequently upon the structure and properties of protective films.

However, in any emulsifier thin intermediate layers of the water phase must indicate a loosening effect (Deryagin /an author/), which also prevents the oil drops from joining. We cannot decide here what the relative importance of the loosening effect is compared with osmotic or electrostatic effects in the stabilization of our systems.

Conclusions

1. Coalescence in highly concentrated oil-water type emulsions protected by sodium oleate was studied.
2. It was found that in emulsions with expanded films the volume coalescence in relation to aging conditions produces either the known decrease in degree of dispersion of the system or its partial breakdown, but in both cases the adsorption layers were concentrated to the point of condensation.
3. In emulsions with condensed films the rate of surface coalescence is incomparably higher than the rate of volume coalescence, as a result of which the system breaks down with aging in the boundary areas (near the free surface or on the border with large deposits).
4. The separation and expansion of emulsions with condensed films of oleate under diverse conditions was investigated.
5. The characteristic of equilibrium and critical layers of the water phase was given.
6. It was proved that bonded water in these systems is maintained by the electrostatic interaction of single oleate layers.

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[Appended tables and figures follow.]

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Table 1. Change of Emulsions in Weak Mixing

Amount of Oleate in mol/l	Ratio	Before Mixing			Ratio	After Mixing		
		$\bar{V} \cdot 10^{-4}$ μ^3	$S \cdot 10^{-3}$ cm^3	O \AA^2		$\bar{V} \cdot 10^{-4}$ μ^3	$S \cdot 10^{-3}$ cm^3	O \AA^2
0.02864	22.5	0.48	42.8	24.8	13.3	0.14	33.0	18.9
0.04296	25.0	0.09	86.8	33.2	18.0	0.17	51.3	19.6
0.05728	31.0	0.07	127.5	36.8	22.0	0.07	79.1	22.8

Table 2. Change of Emulsions With Long Storage

Amount of Oleate in mol/l	Ratio	O \AA^2	Mean Diameter \bar{d} for the Time of Aging in Hours				
			0	144	300	526	650
0.00716	3.8	11	20.7	20.2	21.0	21.5	21.2
0.01432	5.2	11	13.7	12.1	11.7	12.5	12.4
0.02864	12.0	15	12.6	13.0	13.4	13.8	13.0

Table 3. Thickness of Water Layers Under Various Oleate Emulsion Conditions (δ in \AA)

Oil-Water ratio of the emulsion.....	5.16	12	18	32
δ maximum (expansion).....	11 380	4 310	4 200	4 160
δ minimum (separation at 2,000 rpm).....	1 830	750	705	866

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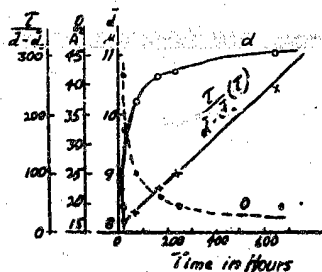


Figure 1. Coalescence in Oleate Emulsions (according to Fisher and Jarkins)

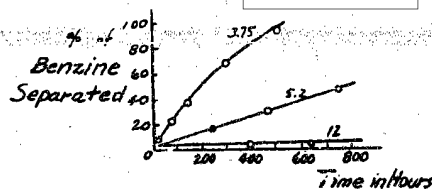


Figure 2. Breakdown of Oleate Emulsions With Aging

The figures on the curves are the oil-water ratio of the emulsion; T is the time in hours; the amount of benzine separated in % of its original content is on the axis of ordinates.

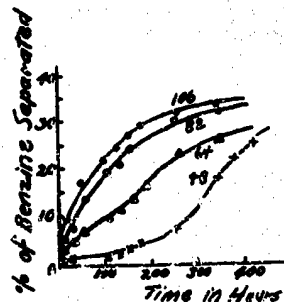


Figure 3. Effect of Size of Container on Breakdown of Emulsion.

Ratio 18:1. The figures on the curves are the diameters of the containers in mm. T is the time in hours.

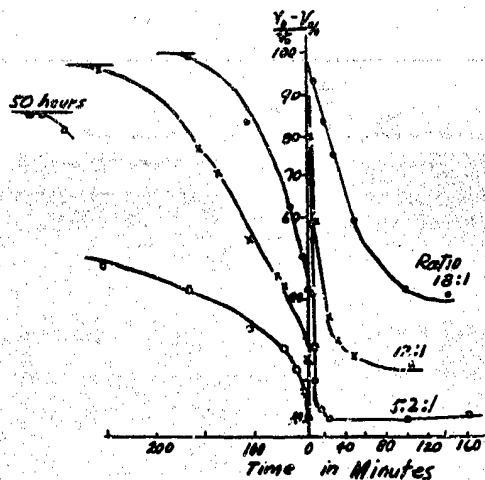


Figure 4. The Energy of Separation (Right) and the Energy of Expansion (Left) of an Emulsion With Condensed Films of Oleate in a Centrifugal Field of 2,000 rpm

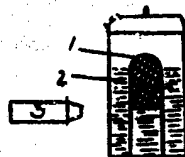


Figure 5. Diagram of Experiment to Determine the Expansion Equilibrium of an Emulsion
1 - emulsion; 2 - water; 3 - cathetometer

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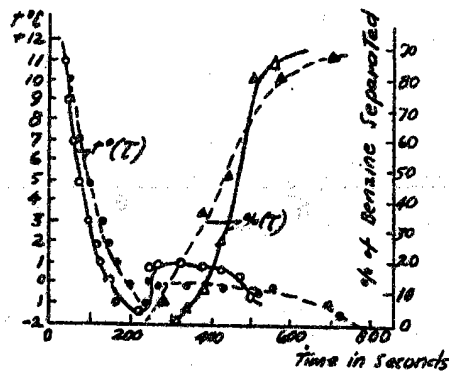


Figure 6. Freezing of Emulsions With Condensed Oleate Layers (-5 degrees C)

- Oleate condensation equal to 0.025 M/liter, ratio 12:1
- Oleate condensation equal to 0.145 M/liter, ratio 32:1

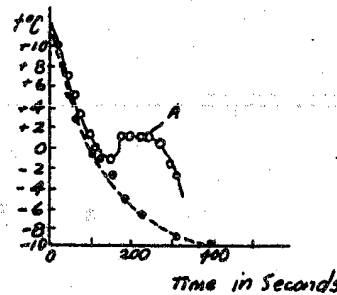


Figure 7. Behavior of Emulsions in Freezing (-12 degrees C)

- Continuous phase water 100%
- Continuous phase: water 27%, glycerine 73%
- A - Complete breakdown of emulsion

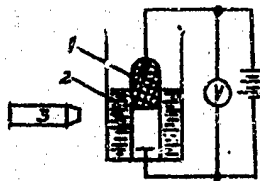


Figure 8. Diagram of Experiment on Cataphoresis of Emulsion

1 - emulsion; 2 - water; 3 - cathetometer

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